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ABSTRACT

Electron-beam melted tungsten-rhenium alloys containing 20 to 28 at. % Re exhibited elongations of over 200 percent in tensile tests at 3630° F (2000° C). Rapid grain growth rates in these alloys, relative to tungsten-rhenium alloys with lesser Re contents, and continuous recrystallization during tensile testing indicate the occurrence of enhanced diffusion processes. Strain-rate sensitivities generally ranged between 0.2 and 0.3 for these alloys, but strain-rate sensitivities as high as 0.8 were observed under the special conditions encountered immediately following initial recrystallization. These were well within the range associated with superplastic materials.

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SUMMARY

The tensile properties of binary tungsten-rhenium alloys containing up to 33-atomic-percent rhenium were determined at temperatures from 78° to 3630° F (25° to 2000° C). Elongations as high as 260 percent were observed in electron-beam melted tungsten containing 23 atomic percent rhenium when tensile tested at 3630° F (2000° C) after a 1-hour anneal at 3090° F (1700° C). All alloys tested under these conditions with rhenium contents between 20 and 28 atomic percent exhibited elongations of at least 200 percent. These alloys also showed enhanced grain growth rates. This resulted in both continual recrystallization and a fivefold increase in grain-growth rate during tensile testing as compared with alloys with rhenium contents less than 20 atomic percent.

The values of strain-rate sensitivity ranged between 0.2 and 0.3 for the alloys exhibiting high elongations. However, for swaged alloys tested just above the recrystallization temperature, strain-rate sensitivities as high as 0.8 were observed. The high temperature strengths of the high-rhenium-content alloys were also less than those of alloys with intermediate rhenium contents.

It thus appears that tungsten alloys containing 22 to 28 atomic percent rhenium may be termed superplastic in the general sense of the term.

INTRODUCTION

Alloying with rhenium (Re) has two distinct effects on the mechanical properties of tungsten (W). One effect occurs in dilute solid solutions and is most clearly seen in the lowering of the hardness and of the ductile-brittle transition temperature at about 5-atomic-percent Re (refs. 1 to 3). The second effect occurs in the concentrated solid solution at about 25-atomic-percent rhenium, and is characterized by twinning and a larger decrease in the ductile-brittle transition temperature.

An aspect of the high-rhenium-content alloy which has received little attention is the higher-than-usual tensile ductility at 3500° F (1927° C) (ref. 3). Anomalous high duc-

tility has been observed in several other alloy systems, usually associated with a phase transformation or a solid solubility limit (refs. 4 and 5). The phenomenon is now commonly termed superplasticity. High strain-rate-sensitivities (ref. 6) and grain boundary sliding (ref. 7) have been reported for materials that undergo superplastic deformation. Also, it has been found that superplastic materials usually have a very fine grain structure (less than 10 μm) (ref. 8), and it has been proposed (ref. 9) that such a structure alone is sufficient for superplasticity. The purpose of the present study was to further characterize the high-temperature tensile properties of tungsten-rhenium alloys to determine the conditions under which they might be superplastic. Tensile and selected strain-rate sensitivity tests were conducted on alloys containing 0- to 33.3-atomic-percent rhenium at temperatures from 78° to 3630° F (25° to 2000° C). Grain-growth rates were also determined on most alloys at 3630° F (2000° C).

Figure 1 indicates the compositions evaluated and their locations relative to the solvus line in the tungsten-rich end of the tungsten-rhenium phase diagram.

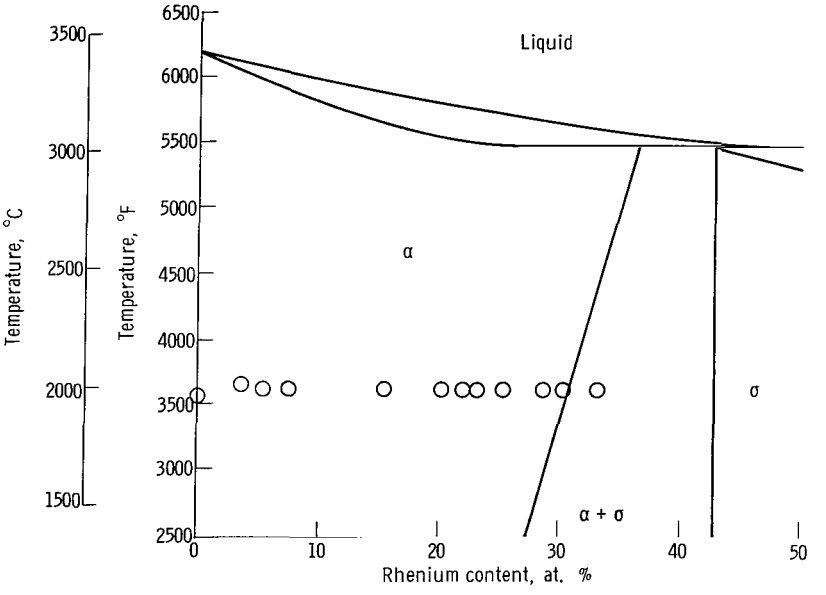


Figure 1. - Tungsten-rich end of tungsten-rhenium phase diagram.

EXPERIMENTAL PROCEDURE

Preparation of Materials

Twelve binary tungsten-rhenium alloys containing up to 33.3-atomic-percent rhenium were double electron-beam melted and subsequently fabricated to 0.3-inch rod by extrusion and swaging. An alloy containing 36-atomic-percent rhenium could not be fabricated.

The starting materials were commercial powders of tungsten and rhenium with impurity levels as listed in table I. The particle size of the tungsten powder averaged 5 micrometers (Fisher number) whereas the rhenium powder was -200 mesh (less than 80 μm). The various blended tungsten-rhenium compositions were hydrostatically pressed at 70 ksi (480 MN/m^2) into 0.8-inch (2-cm) diameter bars weighing approximately 6.2 pounds (2.8 kg).

The bars were melted in a 150-kilowatt commercial electron-beam furnace. Initial consolidation of the unsintered bars by drip melting resulted in 1.5-inch (3.8-cm) diameter ingots. The alloys were then remelted into 2-inch (5-cm) diameter ingots at a

TABLE I. - SUPPLIERS ANALYSES OF STARTING MATERIALS

Impurity element	Impurity content, wt %	
	Tungsten powder	Rhenium powder
Carbon	0.001	0.0059
Oxygen	.024	.27
Hydrogen	-----	.0042
Nitrogen	-----	.0040
Aluminum	<.001	<.001
Calcium	↓	↓
Chromium		
Copper		
Feron		.0008
Potassium		<.0001
Magnesium		.0001
Molybdenum	.003	.0001
Manganese	<.001	N.D.[a]
Sodium	↓	<.0001
Nickel		.0001
Silicon		.0001
Tin		N.D.[a]

[a]Not detected.

TABLE II. - FABRICATION CONDITIONS

Analyzed rhenium content of ingot, at. %	Extrusion temperature[a]		Final swaging temperature[b]	
	^{o}F	^{o}C	^{o}F	^{o}C
0	3200	1760	2300	1260
3.3	3500	1927	[c]2100	1149
5.0	3500	1927	[c]2200	1204
7.7	3800	2093	[c]2100	1149
15.4	4120	2272	2850	1566
20.2	4600	2538	2900	1593
22.0	4000	2204	[c]2600	1427
23.2	4400	2427	2850	1566
25.3	4600	2538	[c]2600	1427
28.7	4600	2538	[c]2600	1427
30.4	4200	2316	[c]2600	1427
33.3	4000	2204	2800	1538

[a]Extrusion reduction ratio, 6:1.
[b]Materials were swaged 83 percent after extrusion.
[c]Initial swaging temperature was 200° F higher.

chamber pressure of 10^{-5} torr (10^{-3} N/m^2) for added purification and improved ingot surface.

The ingots were prepared for extrusion by grinding to 1.75-inch (4.5-cm) diameter billets and canning in unalloyed powder metallurgy molybdenum. The canned billets were induction heated in hydrogen at temperatures ranging from 3200° to 4600° F (1760° to 2537° C), transferred within 4 to 8 seconds to a conventional hydraulic press, and extruded to a round bar at a reduction ratio of 6:1 (table II).

Swaging of the alloy extrusions was performed in the canned condition to about 83-percent total reduction in area at final swaging temperatures ranging from 2100° to 2900° F (1149° to 1593° C). The bars were heated in hydrogen and swaged with about 10-percent reduction per pass to 0.35-inch (0.9-cm) diameter.

Buttonhead tensile specimens were centerless ground from the swaged bars, thereby removing the molybdenum canning. The specimen reduced section had a diameter of 0.16 inch (0.41 cm) and a gage length of 1.0 inch (2.5 cm).

Chemical Analyses

X-ray fluorescence analyses of the alloy tensile bars showed an average loss of 7 percent of the original rhenium content during processing, primarily due to vaporization during melting. Other analyses indicated interstitial impurities ranging from 2 to 7 ppm carbon, 1 to 12 ppm oxygen, and less than 5 ppm nitrogen. Interstitial contents and as-cast hardnesses are listed in table III.

TABLE III. - ANALYSES AND HARDNESSES OF EXPERIMENTAL MATERIALS

Alloy	Rhenium content, at. %	Impurity content, ppm			As-cast Vicker's hardness number
		Oxygen	Nitrogen	Carbon	
EB-193	0	2	--	3	345
EB-212	3.3	2	--	4	322
EB-218	5.0	2	<5	3	306
EB-215	7.7	3	--	3	312
EB-197	15.4	1	--	2	366
EB-198	20.2	6	--	3	416
EB-205	22.0	--	--	--	433
EB-199	23.2	2	<5	2	437
EB-200	25.3	3	--	4	442
EB-201	28.7	6	--	7	446
EB-202	30.4	6	--	7	450
EB-203	33.3	12	<5	6	421

Testing Procedures

Tensile tests were conducted on the buttonhead specimens after electropolishing the gage section to a final diameter of 0.15 inch (0.38 cm). Test temperatures were room temperature, 930°, 1830°, 2730°, and 3630° F (500°, 1000°, 1500°, and 2000° C). The room-temperature tests were performed in air at atmospheric pressure, the 930° F (500° C) tests under vacuum conditions at 5×10^{-2} torr (7 N/m^2), and all other tests at less than 10^{-5} torr (10^{-3} N/m^2). Prior to testing, the tensile specimens were annealed for 1 hour at either 3090° or 4170° F (1700° or 2300° C). Tests were performed in a universal tensile testing machine at a crosshead speed of 0.005 inch per minute (2.1 μm/sec) for specimens annealed at 3090° F (1700° C) and 0.01 inch per minute (4.2 μm/sec) for those annealed at 3630° F (2000° C). Because of the limited length of

the resistance heater used in tensile testing (7 in. (17.5 cm)), nonuniform temperature conditions undoubtedly existed in those tests where elongations approached or exceeded 200 percent. However, the elongations measured were probably less than those which would have occurred in the gage lengths of uniformly heated specimens.

Strain-rate sensitivity tests were conducted in a universal testing unit adapted to permit rapid changes of strain rate (crosshead speed) by electromechanical means over several orders of magnitude. During each test, the strain rate was changed in discrete steps between 0.002 and 2.0 inches per minute (0.84 and 840 μm/sec). These tests were conducted at 3630° F (2000° C). From these test data, the true-stress true-strain curves were reconstructed for each strain rate, assuming constant volume and uniform elongation. The strain-rate sensitivity m defined as

$$m = \frac{d \ln \sigma}{d \ln \dot{\epsilon}}$$

where σ is the stress and $\dot{\epsilon}$ the strain rate, was then determined for each alloy from the slope of a flow stress against strain rate plot.

Grain growth rates of the swaged alloys were determined from specimens annealed for 1/2 to 3 hours at 3630° F (2000° C). After each 1/2-hour anneal, portions of each specimen were polished and etched, and grain size measurements were made by the line intercept method (ref. 10).

The nonequiaxiality of grains was also measured at the fracture zones of some specimens after testing. The nonequiaxiality is expressed as the ratio of grain width to grain length, as determined by a straight line intercept technique.

RESULTS

Tensile Properties

An initial survey of the tensile properties of the alloys as a function of rhenium content was performed on specimens annealed at 4170° F (2300° C) for 1 hour. The treatment resulted in a fully recrystallized equiaxed grain structure. The results of these tests are summarized in figure 2 and table IV.

At temperatures between 930° and 2730° F (500° and 1500° C), additions of rhenium had only moderate effects on ductility. However, at 3630° F (2000° C) rhenium greatly increased the ductility of tungsten. From a value of 60 percent for unalloyed tungsten, the elongation increased to 185 percent for the W - 25.3-atomic-percent Re alloy. Such an anomalous increase in ductility with temperature has been observed in the chromium-cobalt and the chromium-ruthenium systems by Stephens and Klopp (ref. 11). The very

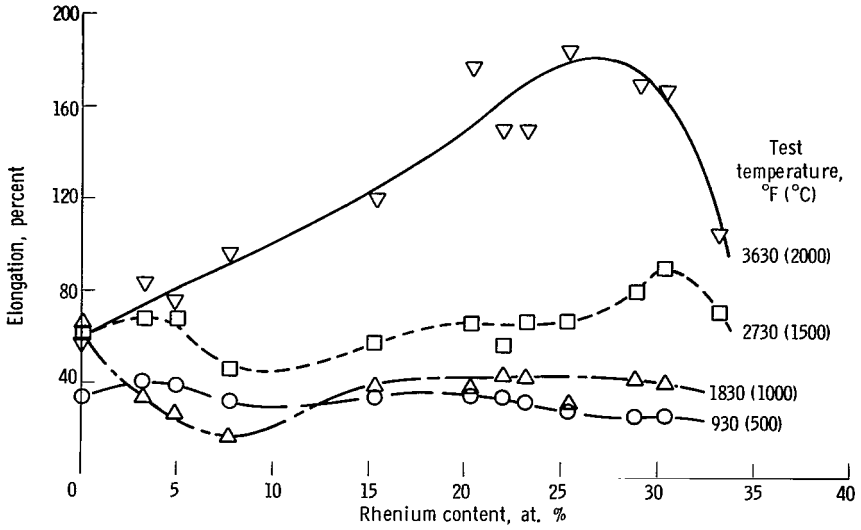


Figure 2. - Effect of test temperature on tensile elongation of tungsten-rhenium alloys annealed 1 hour at 4180° F (2300° C).

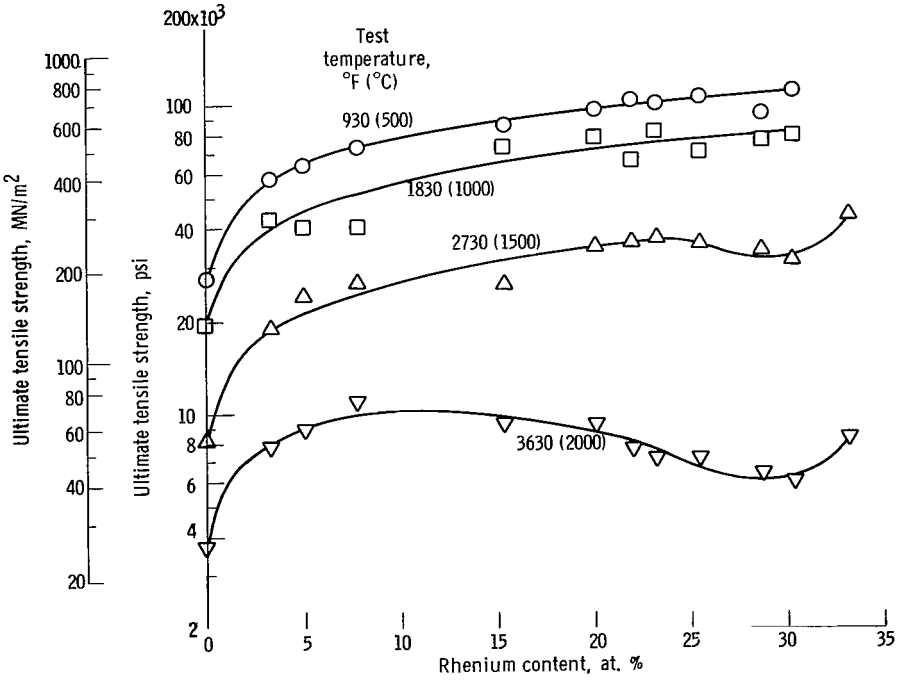


Figure 3. - Effect of test temperature on tensile strength of tungsten-rhenium alloys annealed 1 hour at 4170° F (2300° C).

TABLE IV. - TENSILE DATA FOR TUNGSTEN-RHENIUM ALLOYS RECRYSTALLIZED FOR 1 HOUR AT 4170° F (2300° C)[a]

Specimen	Specimen composition, at. %		Test temperature		Proportional limit stress		Ultimate tensile stress		Elongation, percent	Reduction in area, percent	Button-head average grain diameter, μm	Reduced section	
	Tungsten	Rhenium	°F	°C	ksi	MN/m²	ksi	MN/m²				Grain shape factor	Average grain, diameter, μm
193	100	----	78	25	23.8	164	23.8	164	2	0	2060	----	---
			930	500	~8.5	58.6	27.5	190	33	>99	2670	----	---
			1830	1000	~1.5	10.3	19.3	133	65	↓	2780	----	---
			2730	1500	~1.5	10.3	8.2	56.5	60	↓	1370	----	---
			3630	2000	-----	-----	3.7	25.5	60	↓	5670	1.0	652
212	96.7	3.3	78	25	84.4	582	84.4	582	2	1	178	----	---
			930	500	30.8	212	58.6	404	40	46	151	----	---
			1830	1000	13.4	92.3	42.5	293	32	87	180	----	---
			2730	1500	9.0	62.0	18.9	128	68	>99	166	----	---
			3630	2000	4.6	31.7	7.8	53.8	82	>99	182	0.50	304
218	95.0	5.0	78	25	58.4	403	58.4	403	0	0	173	----	---
			930	500	30.9	213	64.2	443	39	51	208	----	---
			1830	1000	17.5	120	40.2	277	27	78	195	----	---
			2730	1500	13.4	92.3	23.8	164	68	90	185	----	---
			3630	2000	5.0	34.5	8.7	60.0	76	>99	210	0.38	357
215	92.3	7.7	78	25	60.9	420	60.9	420	1	0	179	----	---
			930	500	42.0	290	72.9	503	32	40	186	----	---
			1830	1000	17.5	120	40.0	276	17	6	266	----	---
			2730	1500	16.5	114	26.5	183	48	60	178	----	---
			3630	2000	5.8	40.0	10.8	74.5	95	>99	195	0.50	271
197	84.6	15.4	78	25	74.1	511	83.2	574	1	0	----	----	---
			930	500	51.9	358	86.2	594	33	42	201	----	---
			1830	1000	~40.0	276.0	73.0	503	39	75	196	----	---
			2730	1500	16.3	112.0	26.1	180	59	80	157	----	---
			3630	2000	5.8	40.0	9.2	63.4	120	>99	195	0.50	82
198	79.8	20.2	78	25	62.5	431	62.5	431	2	0	----	----	---
			930	500	52.5	362	96.5	665	34	62	177	----	---
			1830	1000	~50.0	345	78.0	538	38	84	222	----	---
			2730	1500	22.5	155	~34.5	238	65	89	202	----	---
			3630	2000	6.3	44.8	9.1	62.7	177	>99	219	0.65	42

[a]Crosshead speed, 0.01 in./min (4.2 μm/min).

TABLE IV. - Concluded. TENSILE DATA FOR TUNGSTEN-RHENIUM ALLOYS RECRYSTALLIZED FOR 1 HOUR AT 4170° F (2300° C)[a]

Specimen	Specimen composition, at. %		Test temperature		Proportional limit stress		Ultimate tensile stress		Elongation, percent	Reduction in area, percent	Button-head average grain diameter, μm	Reduced section	
												Grain shape factor	Average grain diameter, μm
	Tungsten	Rhenium	°F	°C	ksi	MN/m²	ksi	MN/m²					
205	78.0	22.0	78	25	65.4	451	73.0	503	2	0	---	----	---
			930	500	67.1	463	103.0	710	32	61	227	----	---
			1830	1000	41.1	283	65.5	452	42	83	218	----	---
			2730	1500	23.1	159	35.4	244	58	87	180	----	---
			3630	2000	5.7	39.3	7.6	52.4	150	>99	217	0.71	41
199	76.8	23.2	78	25	~60.0	414	74.5	514	2	0	---	----	---
			930	500	~60.0	414	100.1	690	31	60	209	----	---
			1830	1000	50.0	345	81.1	559	41	87	214	----	---
			2730	1500	24.8	171	37.1	256	67	87	178	----	---
			3630	2000	2.5	17.2	7.1	49.0	150	>99	234	1.00	41
200	74.7	25.3	78	25	89.5	617	89.5	618	2	0	---	----	---
			930	500	74.6	514	105.5	727	29	44	221	----	---
			1830	1000	43.9	303	70.5	486	30	88	279	----	---
			2730	1500	23.7	163	35.6	245	68	94	225	----	---
			3630	2000	4.9	33.8	7.1	49.0	185	>99	282	1.00	58
201	71.3	28.7	78	25	51.0	352	102.0	703	4	3	---	----	---
			930	500	63.0	434	93.5	645	26	53	245	----	---
			1830	1000	52.1	359	76.0	524	43	80	300	----	---
			2730	1500	26.2	181	33.3	230	80	>99	234	----	---
			3630	2000	4.8	33.1	6.3	43.4	170	>99	316	0.74	107
[b]202	69.6	30.4	78	25	52.6	363	112.0	772	5	2	---	----	---
			930	500	58.1	401	109.8	757	26	70	---	----	---
			1830	1000	49.1	339	78.9	544	40	80	271	----	---
			2730	1500	24.5	169	31.3	216	91	94	278	----	---
			3630	2000	5.2	35.9	6.0	41.4	168	>99	304	0.67	---
[b]203	66.7	33.3	78	25	84.0	579	84.0	579	1	1	---	----	---
			2730	1500	20.5	141	43.5	300.0	72	66	---	----	---
			3630	2000	5.2	35.9	8.3	57.2	105	70	---	1.00	---

[a]Crosshead speed, 0.01 in./min (4.2 μm/min).

[b]Alloy contained some sigma phase.

TABLE V. - TENSILE DATA FOR TUNGSTEN-RHENIUM ALLOYS

AT 3630° F (2000° C)[a]

Alloy composition	Proportional limit stress		Ultimate tensile stress		Elongation, percent	Reduction in area, percent	Buttonhead average grain diameter, μm
	ksi	MN/m^2	ksi	MN/m^2			
W-7.7 Re	6.09	42.1	8.41	57.9	135	---	113
W-15.4 Re	6.08	42.0	8.33	57.2	179	>99	99
W-20.2 Re	5.98	41.4	6.98	48.3	210	---	122
W-22.0 Re	5.56	38.6	6.64	45.5	262	>99	168
W-23.2 Re	5.98	41.4	6.37	44.1	218	↓	159
W-25.3 Re	5.42	37.2	6.39	44.2	254	↓	207
W-30.4 Re	5.55	38.5	7.57	52.4	233	↓	---
W-33.3 Re	3.58	24.8	5.56	38.6	135	67	---

[a]Materials were annealed for 1 hr at 3090° F (1700° C) prior to testing. Crosshead speed, 0.005 in./min (2.1 μm/sec).

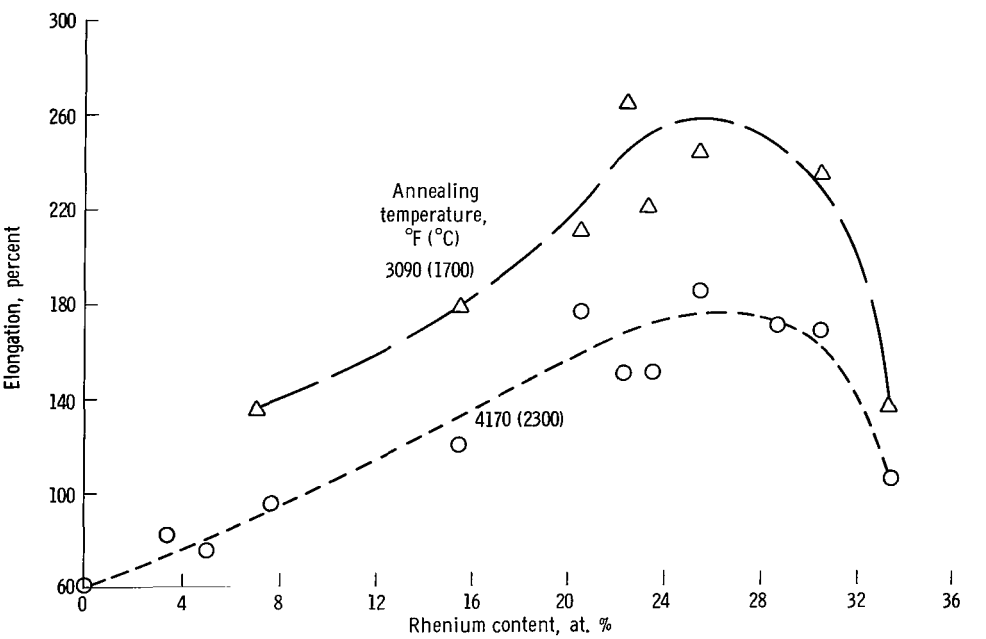


Figure 4. - Effect of annealing temperature on ductility of tungsten-rhenium alloys. Tensile elongation measured at 3630° F (2000° C).

high elongations at 3630° F (2000° C) for tungsten alloys containing 20- to 30-atomic-percent rhenium is reflected in the tensile strengths, which exhibit a distinct minimum in the vicinity of 28-atomic-percent rhenium (fig. 3).

To determine the effects of annealing temperature on the tensile properties, specimens were annealed at 3090° F (1700° C) for comparison with those annealed at 4170° F (2300° C). The results of these tests, which were conducted at a strain rate of 0.01 inch per minute (4.2 μm/sec) rather than 0.005 inch per minute (2.1 μm/sec), are summarized in table V. The elongations of these specimens as a function of rhenium are compared in figure 4. As a result of the lower annealing temperature, the maximum elongation was increased from 185 to 262 percent. In light of this behavior, the effect of annealing temperature was further investigated.

Figure 5 illustrates the effect of various annealing temperatures on both grain size and tensile elongation of the W - 25.3-percent-Re alloy. The results of these tensile

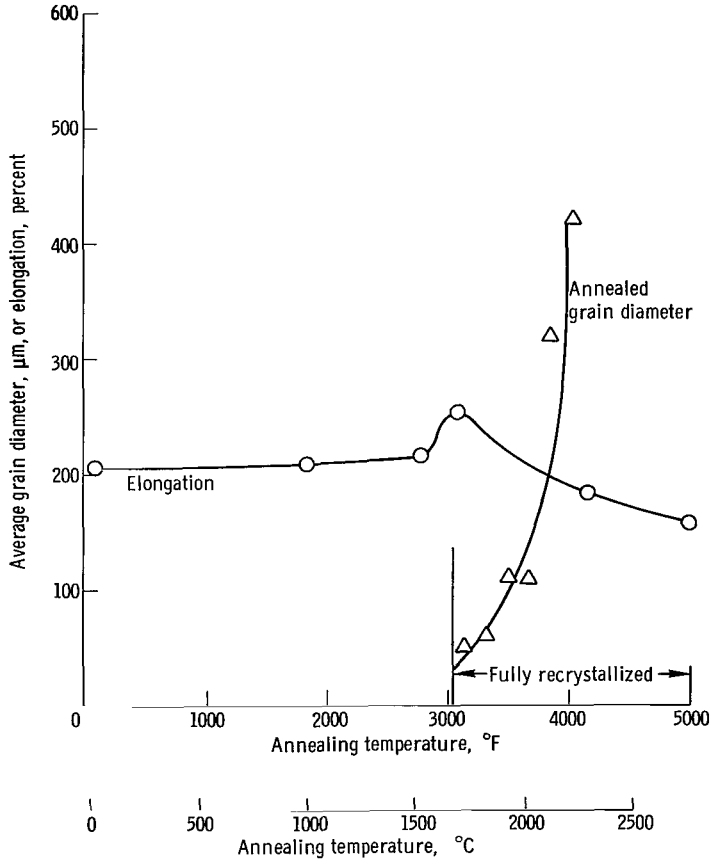


Figure 5. - Annealed grain size for tungsten-25.3-atomic-percent-rhenium specimens annealed 1 hour and elongation of annealed specimens pulled at 3630° F (2000° C).

TABLE VI. - TENSILE PROPERTIES OF TUNGSTEN - 25.3-ATOMIC-PERCENT RHENIUM ALLOY AT 3630° F (2000° C)

[Reduction in area, >99 percent.]

Annealing temperature[a]		Proportional limit stress		Ultimate tensile stress		Elongation, percent
°F	°C	ksi	MN/m^2	ksi	MN/m^2	
(b)	(b)	5.60	38.6	6.85	46.9	205
1830	1000	5.16	35.9	6.19	42.7	210
2780	1530	5.70	39.3	6.44	44.1	215
3090	1700	5.42	37.2	6.39	44.0	254
[c]4170	[c]2300	4.90	33.8	7.10	49.0	185
5000	2760	5.50	37.9	6.46	44.8	158

[a]Annealing time, 1 hr.

[b]As-swaged.

[c]Crosshead speed, 0.01 in./min (4.2 μm/sec). All other tests were 0.005 in./min (2.1 μm/sec).

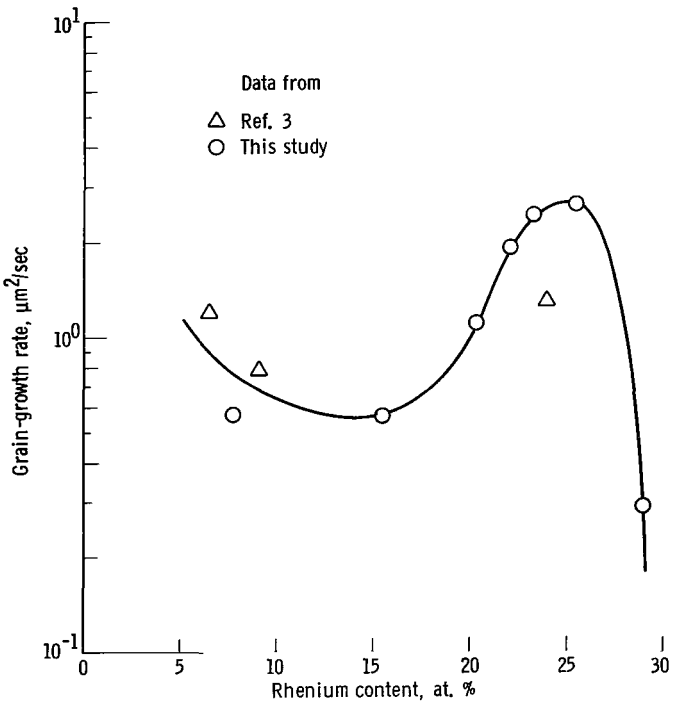


Figure 6. - Grain-growth rates of tungsten-rhenium alloys at 3630° F (2000° C).

TABLE VII. - GRAIN SIZES OF TUNGSTEN-RHENIUM ALLOYS ANNEALED AT 3630° F (2000° C)

Rhenium content, at. %	Duration of anneal, hr					Average grain-growth rate, $\mu m^2/sec$
	0.5	1.0	1.5	2.0	3.0	
	Average grain diameter, μm					
7.7	--	65	---	73	91	0.56
15.4	--	60	---	75	88	.56
20.2	54	75	83	110	130	1.1
20.2	--	71	---	84	93	
22.0	59	77	93	120	130	1.9
22.0	--	79	---	100	140	
22.0	67	92	100	120	150	
23.2	49	73	91	120	160	2.4
23.2	--	74	---	87	120	
25.3	74	110	120	140	170	2.6
25.3	--	88	---	130	160	
28.7	41	43	73	45	55	.29
30.4	(a)	(a)	(a)	(a)	(a)	----
33.3	(a)	(a)	(a)	(a)	(a)	----

[a]Two phase microstructure. Not fully recrystallized.

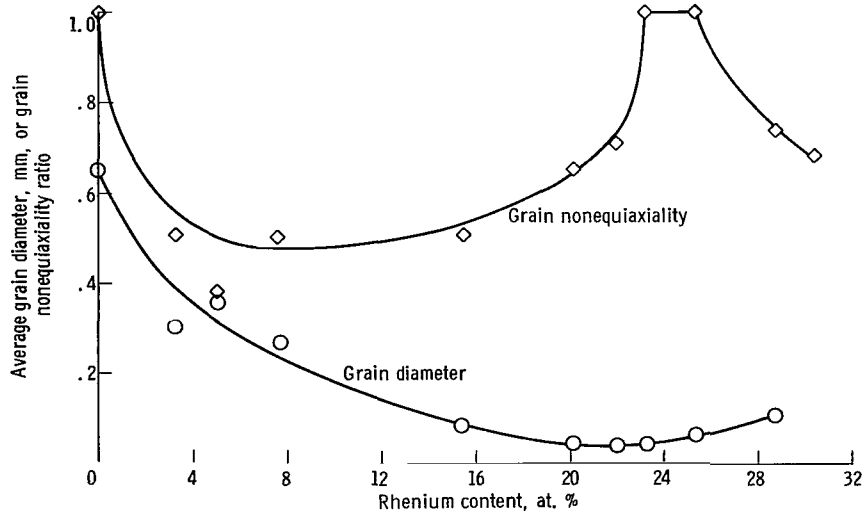
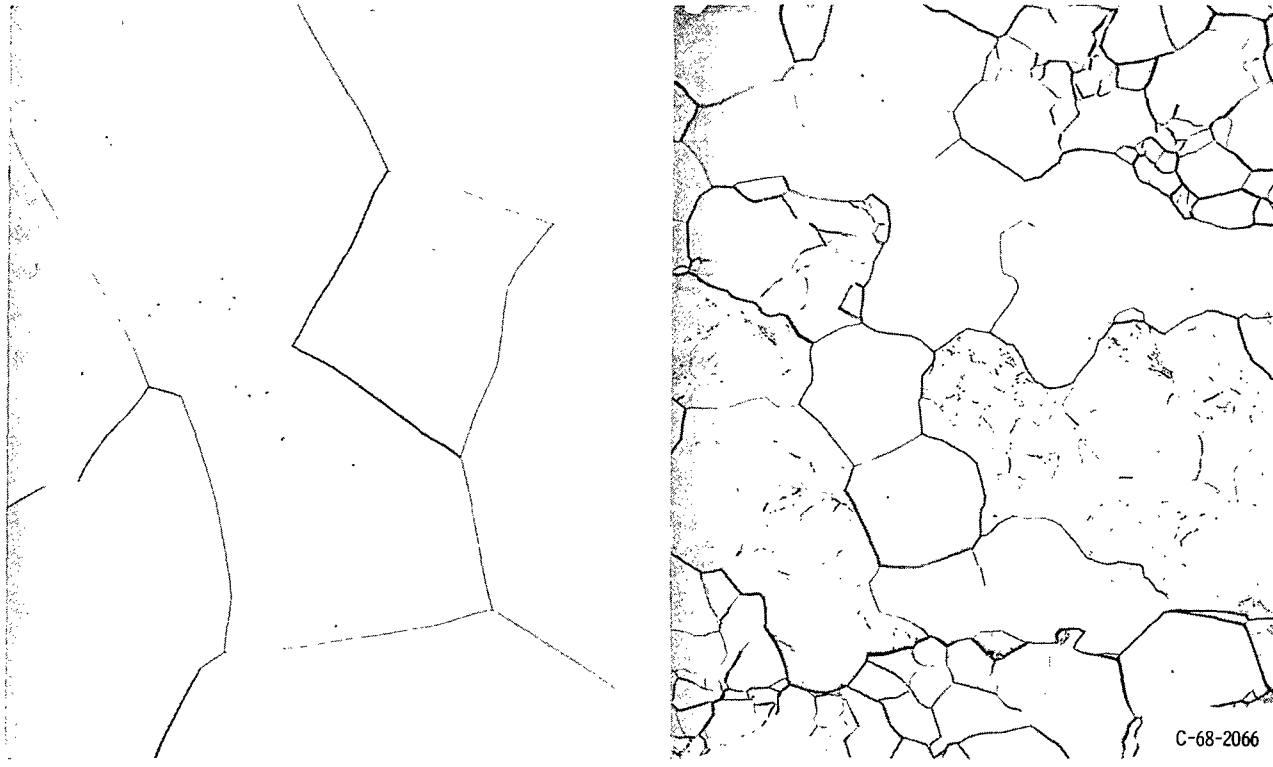


Figure 7. - Average grain diameter and grain nonequiaxiality in fracture zone of tungsten-rhenium alloys. Test temperature, 3630° F (2000° C).



(a) Buttonhead; average grain diameter, ≈ 300 micrometers.

(b) Fracture zone; average grain diameter, ≈ 50 micrometers.

Figure 8. - Microstructure of a tungsten - 23.2-atomic-percent rhenium tensile specimen pulled at 3630° F (2000° C) after a 1 hour anneal at 4170° F (2300° C). X250.

tests are summarized in table VI. The highest elongation at the test temperature 3630° F (2000° C) occurred for specimens annealed at 3090° F (1700° C). The effect of annealing temperature on the recrystallized grain size is also illustrated. The grain size increased logarithmically with annealing temperature in the conventional manner.

Grain-Size Studies

At the low crosshead speeds used in this study (0.005 or 0.01 in./min (2.1 or 4.2 μm/sec)), the duration of the tensile tests at 3630° F (2000° C) often exceeded 5 hours, so that grain growth rates are probably more important than annealed grain size prior to testing in determining the structures. Therefore, a limited study of grain-growth rates was undertaken.

Grain-size measurements were made at various time intervals on unstressed specimens heated at 3630° F (2000° C). The square of the grain diameter varied linearly with elapsed time, indicative of a parabolic grain-growth rate. Figure 6 illustrates the grain-growth rate at 3630° F (2000° C) as a function of rhenium content. A sharp maximum occurs at composition levels where the maximum elongation was also observed. The grain-size data from which the rates were calculated are also given in table VII.

The average grain diameter and the degree of grain nonequiaxiality in the fracture zone after testing at 3630° F (2000° C) are illustrated in figure 7. The grain diameter is at a minimum at W - 23.2-percent Re where essentially equiaxed grains were observed. This structure is shown in figure 8, along with the grain structure in the buttonhead. The grain size in the fracture area is considerably smaller than in the buttonhead.

Strain-Rate Effects

Strain-rate sensitivities were determined by changing the crosshead speed in increments during tensile testing. The specimens were pulled at 3630° F (2000° C) after a 1-hour anneal at 3090° F (1700° C). For all specimens examined, the load at any particular crosshead speed decreased continuously with increasing strain from its value at yielding. The calculated true-stress values are relatively constant with increasing strain (fig. 9). The strain-rate sensitivities were computed from such curves for each alloy and are illustrated in figure 10.

The strain-rate sensitivity for all of the alloys near the solubility limit ranged between 0.25 and 0.29 and showed very little variation with strain rate. The W - 33.3-percent-Re specimen did, however, exhibit a significant strain-rate sensitivity with

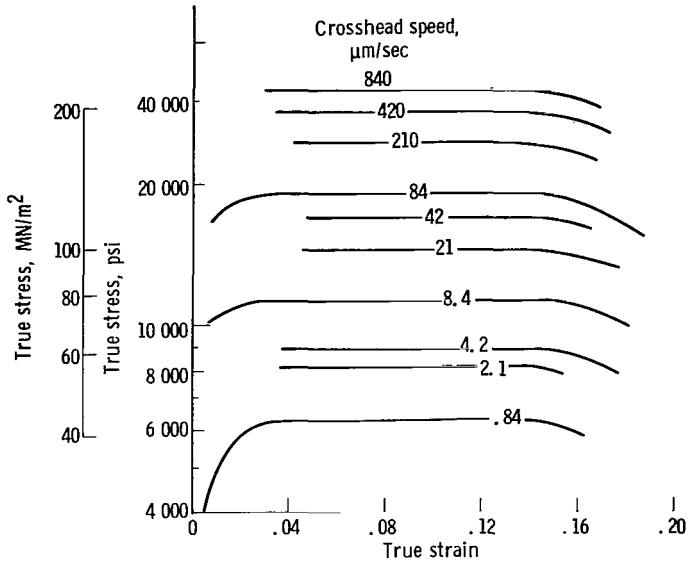


Figure 9. - True-stress true-strain diagram constructed from chart data obtained by continually varying crosshead speed. Tungsten-22.0-atomic-percent rhenium tensile specimen was pulled at 3630° F (2000° C) after a 1 hour anneal at 3090° F (1700° C).

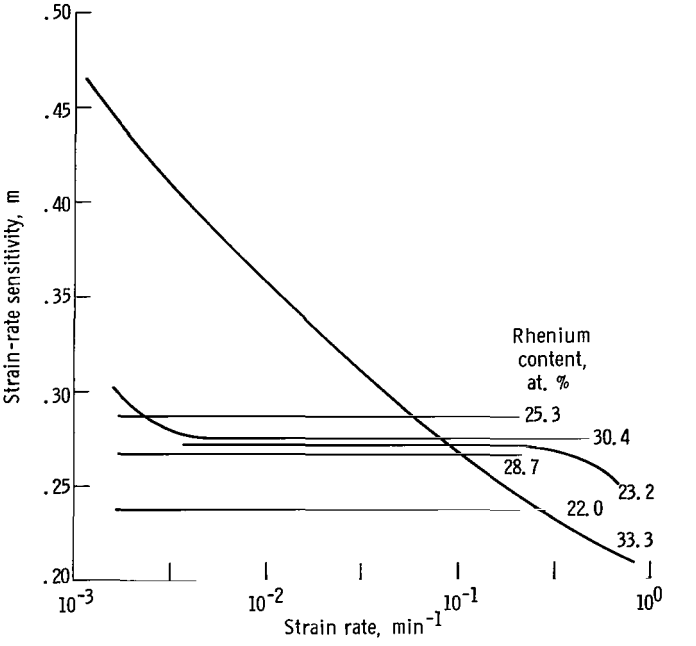


Figure 10. - Strain-rate sensitivity of various tungsten-rhenium alloys at 3630° F (2000° C) after annealing 1 hour at 3090° F (1700° C).

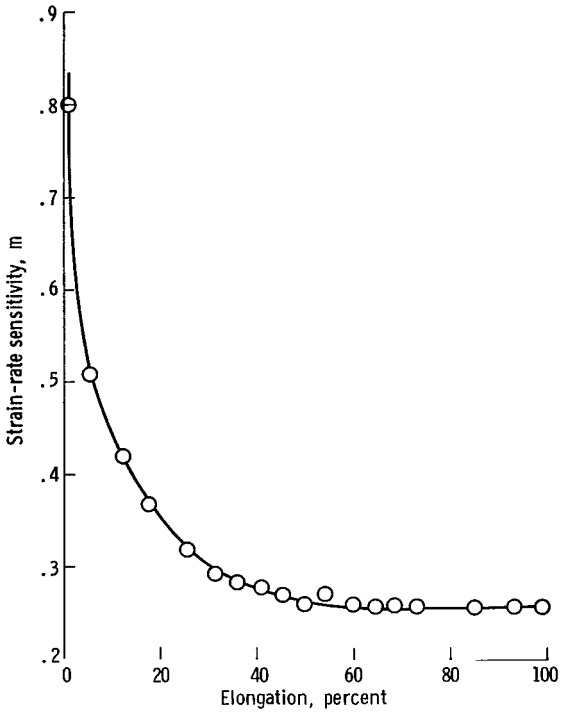


Figure 11. - Strain-rate sensitivity as function of strain for as-swaged tungsten-22.0-atomic-percent rhenium specimen pulled at 3630° F (2000° C).

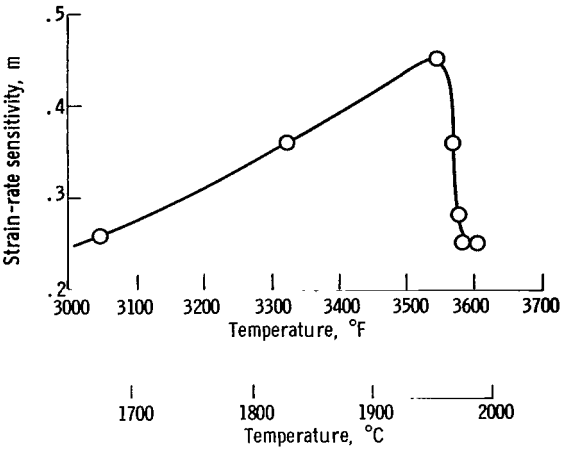


Figure 12. - Strain-rate sensitivity as function of test temperature for tungsten-23.2-atomic-percent rhenium tensile specimen.

strain rate, decreasing from 0.46 to 0.22 with an increase in strain rate of three orders of magnitude.

The strain-rate sensitivities are highly dependent on test conditions. Figure 11 illustrates the change of strain-rate sensitivity with strain for an as-swaged W - 22.0-percent-Re specimen. After rapid heating to 3630° F (2000° C), the specimen was immediately tensile tested while the crosshead speed was shifted between 0.005 and 0.05 inch per minute (2.1 and 21 μm/sec). In figure 12, an as-swaged W - 23.2-percent-Re specimen was heated to 3630° F (2000° C) while it was being pulled. The crosshead speed was shifted between 0.005 and 0.05 inch per minute during testing. Strain-rate sensitivities as high as 0.8 and 0.45 can be seen in figures 11 and 12, respectively.

DISCUSSION

Superplastic behavior is generally associated with either allotropic or other transformation processes (ref. 12) or with fine grain structures (ref. 13). Mechanistically, fine grain, (<10 μm) superplastic behavior has been attributed to viscous deformation processes (ref. 14), grain-boundary shear (ref. 15), or diffusion controlled structural

changes, such as concurrent recrystallization (ref. 6).

Only under conditions of very fine grain size (<1 μm) and high strain-rate sensitivity ($m \approx 1$) could viscous creep accommodate the observed elongation. These conditions were not observed. During testing, strain-rate sensitivities ranged between 0.2 and 0.3, and grain sizes were at least 50 micrometers.

If it is assumed that grain boundaries exhibit viscous shear behavior, grain boundary shear as a contributing mechanism along with grain deformation may explain the high strain-rate sensitivities associated with superplastic behavior. Unfortunately, grain boundary triple junctions act as obstacles to boundary shear, restricting such motion. High grain-boundary mobility can relieve triple-junction stresses, but in single phase materials, such mobility would probably also result in high grain-growth rates, decreasing grain-boundary area. Virtually all fine grain superplastic studies have been restricted to two-phase materials in which grain growth has been a minor factor.

Concurrent recrystallization during tensile testing could be a major factor in permitting grain-boundary shear to occur in single-phase materials. Such a process could maintain a fine grain structure while allowing the high grain-boundary mobility necessary to relieve stresses at triple junctions.

Figure 6 illustrates high-grain-growth rates in the unstressed high-rhenium-content alloys, which indicates enhanced grain-boundary mobility. Figure 8 illustrates that grains in the fracture zone of one of these alloys are significantly smaller than those in the buttonhead after tensile testing, indicating concurrent recrystallization. Apparently, the enhanced diffusional processes evident in the high-rhenium-content alloys can result in either high growth rates or ease of recrystallization. In unstressed specimens, the high grain-growth rate predominates, resulting in a large grain structure, while in tensile specimens, recrystallization processes predominate, resulting in a small grain structure.

Generally, direct measurements of grain-boundary shear consist of either measuring the displacement across grain boundaries of lines on the surface of tensile specimens or measurement of grain nonequiaxiality after deformation. The first method suffers from dependence on surface deformation. The system of mechanical restraints affecting deformation processes on the surface of a metal is different than that at the interior, so that the degree of surface-boundary shear may not be directly related to the amount of grain-boundary shear within the specimen.

Grain shape can indicate the degree of grain deformation occurring, and thus the extent of specimen elongation attributed to grain deformation as opposed to grain-boundary shear. Equiaxed grains persisting after extensive tensile deformation would indicate that the primary mode of deformation was boundary shear, providing that diffusional processes, such as recrystallization and grain growth, which could change grain topography, were absent. As this was not the case in this study, nor probably for any mate-

rial deformed above half its melting temperature, grain equiaxiality data can offer only a qualitative indication of the deformation processes involved in superplastic behavior. Nevertheless, the presence of equiaxed grains in the fracture zone indicates that grain-boundary shear and concurrent recrystallization are probably both occurring. Such a process has been suggested by Holt and Backofen (ref. 15) and by Holt (ref. 16), who reported that the contribution of grain-boundary shear was significant during the deformation of aluminum-zinc alloys.

Although it is not yet possible to identify the deformation processes that distinguish the superplastic materials from their more conventional counterparts, it is possible to compare these materials to determine whether there is a logical division between superplastic and conventional materials.

The tungsten-rhenium alloys near the solubility limit, with strain-rate sensitivities between 0.2 and 0.3, exhibited elongations between 200 and 300 percent. Interestingly, this range of elongations has also been observed by other investigators, listed in figure 13, for materials with strain-rate sensitivities between 0.2 and 0.3. Thus, it would be instructive to compare the elongations of as many materials as possible as a function of strain-rate sensitivity.

Figure 13 illustrates primarily the data of Lee and Backofen (ref. 8) as presented in their figure 6, revised so that specimen extension is expressed as true fracture strain instead of percent elongation. The data of Alden (ref. 17) and Klopp, Witzke, and Raffo (ref. 3), as well as additional data of references 6 and 9, are presented along with the results of this study. Data for unalloyed plutonium are also shown (refs. 18 and 19). The data of Klopp are for tungsten-rhenium alloys which show no superplastic behavior; nevertheless, the alloys with the highest strain-rate sensitivities showed the greatest elongation. Although there is considerable data scatter in figure 13, this figure certainly shows a close relation between fracture strain and strain-rate sensitivity, regardless of the alloy system involved, or whether the superplastic behavior is attributed to transformation processes or to microstructure. This is not to suggest that a high strain-rate sensitivity alone is sufficient for a material to exhibit high elongation, but that it is a necessary condition. Some oxides are known (ref. 20) to have high strain-rate sensitivities, but have limited elongations.

Figure 13 also indicates that to distinguish certain alloys as superplastic and others as not is strictly arbitrary. Certain alloys illustrated have true fracture strains ranging from less than 0.5 to greater than 2.0, depending on test conditions. This suggests that superplastic behavior is more a function of microstructure than alloy composition, as is suggested by figures 11 and 12. Probably any alloy could show high elongations if the test conditions were such as to promote the formation and retention of an extremely fine grain structure during the course of the test. This contention is supported by work on unalloyed cold-worked nickel (ref. 21) tested above the recrystallization temperature

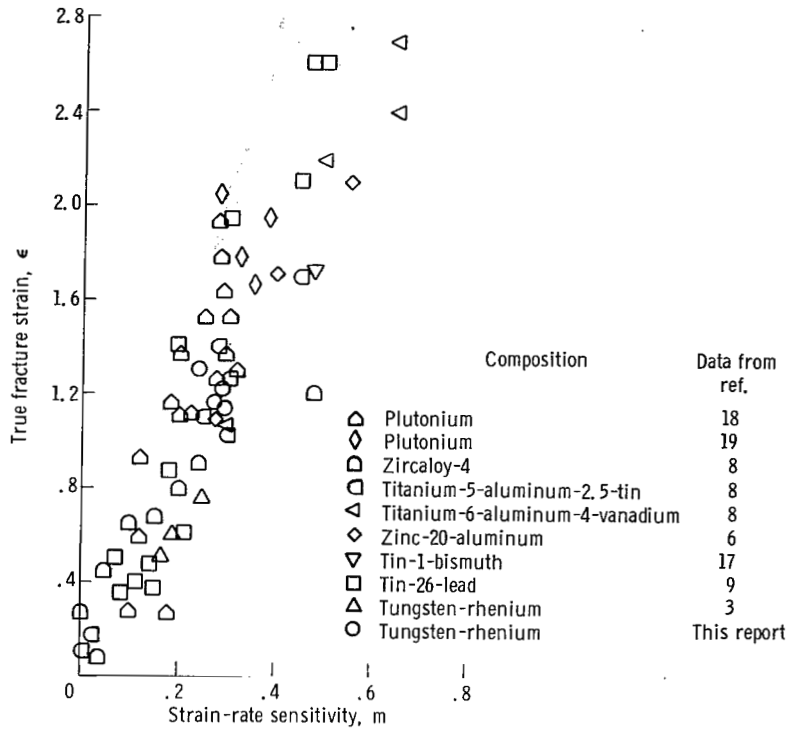


Figure 13. - Fracture strain as function of strain-rate sensitivity for number of alloys tested above one half their melting temperatures.

where a variation of 10 seconds on holding time before testing resulted in a twofold change in total elongation. This single-phase material, when tested in the critical period immediately following the initiation of recrystallization, when the grain size was undoubtedly very fine, showed elongations up to 150 percent. When held 10 seconds longer, however, permitting grain growth to occur, elongations of only 75 percent were observed. Alloys in which fine-grain superplasticity has been observed are commonly two phase. The presence of the second phase is believed to inhibit grain growth so that the initial fine-grain size is maintained during testing. Probably, if the optimum test conditions could be found to maintain the fine-grain size associated with high strain-rate sensitivities in the single phase tungsten-rhenium alloys during testing, these alloys would exhibit greater elongations. Nevertheless, under the conditions observed, elongations up to 260 percent were observed.

CONCLUSIONS

The following conclusions have been drawn from a study of high-temperature tensile properties of tungsten-rhenium alloys:

1. In comparison with other tungsten-base alloys, which rarely exhibit over 80-percent elongation, the high ductilities (up to 260 percent at 3630° F (2000° C)) of tungsten alloys containing approximately 25-atomic-percent rhenium must be considered anomalous, and suggest a difference in deformation mechanism.

2. The equiaxiality of grains in the deformed section of the tensile specimens exhibiting high elongations suggests that grain-boundary sliding contributes significantly to deformation. This conclusion is reinforced by the observation of high grain-boundary mobility, which tends to relieve stresses at grain-boundary triple junctions.

3. Because microstructure and test conditions appear to have as great an effect on strain-rate sensitivity as does alloy composition, and, therefore, on total elongation, designating an alloy as being superplastic is arbitrary. It is probable that many alloys not generally considered superplastic would show very high elongations if evaluated under appropriate test and structural conditions.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 22, 1968,
129-03-02-02-22.

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